THE NICKEL BULLETIN

A SUMMARY OF CURRENT INFORMATION ON NICKEL

DECEMBER, 1953

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Analysis and Determination of Metals and Alloys

R. S. YOUNG: 'Industrial Inorganic Analysis.' Published by Chapman and Hall, Ltd., London, 1953; 368 pp. Price 36/-.

The author points out in his preface that although many good text books on analytical chemistry are readily available, such compilations tend to omit consideration of many elements which must be included in the work of a general inorganic analytical laboratory, and that in other cases little guidance is given as to practical experience which would assist a chemist in selecting the best method for use in given conditions.

To fill these gaps, the material in the present book has been compiled from notes accumulated over several years in the course of practical work in a typical laboratory. It includes brief discussion of theory in relation to the respective methods considered, recommendations on the separations necessary in dealing with a complex sample (and, frequently, on the required manipulative technique), with a critical appraisal, on the basis of the author's experience, of the value of the various methods for general or highly specialized work. The manual is therefore of an essentially practical character, designed for dual service: to the works' chemist, and to the student of graduate or equivalent level who wishes to become familiar with the technique of the industrial chemist and his mode of approach to analytical problems.

Analysis and estimation of forty-three elements are individually covered, and a final chapter contains information on miscellaneous analysis: acidity of special samples; gas analysis; analysis of water; determination of xanthates; preparation of standard acids, bases and indicators.

A *useful list of reference books* on industrial inorganic analysis is appended and each chapter is supported by a selected *bibliography*.

The chapter on Nickel contains sections dealing with determination of nickel (1) with dimethylglyoxime; (2) volumetrically with potassium cyanide, and (3) electrolytically.

Some special procedures, suitable, for example, for determination of very small quantities of nickel, are also included.

Shell Moulding of Stainless Steel

See abstract on p. 214.

NICKEL

Electrical Conductances of Thin Evaporated Nickel Film

N. MOSTOVECH: 'Experimental Study of the Electrical Conductances of Very Thin Metallic Deposits obtained by Thermal Evaporation.' *Annales de Physique*, 1953, vol. 8, pp. 61–125.

Films, less than 100 Å in thickness, were deposited on glass, quartz, or mica, by thermal evaporation, and their resistances were determined. The metals studied were tungsten, molybdenum, rhodium, platinum, iron, nickel, copper, gold and aluminium. Results are well summarized in *Chemical Abstracts*, 1953, vol. 47, Sept. 10, p. 8446.

Ceramic Coating of Nickel for High-Temperature Service

'Ceramic Coating Applied to Nickel.' *Materials and Methods*, 1953, vol. 38, Oct., p. 240.

Work at the Oak Ridge U.S. National Laboratory has shown that adherent and durable ceramic coatings may be deposited on nickel which, before being sprayed, has been annealed in water-saturated hydrogen at about 1000°F. (540°C.).

Tests up to 65 hours in duration, made at 1500°F. (815°C.), on coated specimens, showed good adherence and unimpaired appearance of the coating. Exposure, in similar conditions, of specimens ceramic-coated after dry-hydrogen annealing of the nickel resulted in bubbly surfaces and rapid deterioration of the coating bond. The ceramic used in the tests was the A.418 type developed at the National Bureau of Standards. No details of its composition are given. The process may be expected to extend the use of nickel components in high-temperature oxidizing conditions such as those encountered in gas turbines.

Radiation Properties of Nickel-containing Materials at Elevated Temperatures

See abstract on p. 212.

Measurement of Internal Stresses in Nickel Wires

U. DEHLINGER and H. SCHOLL: 'Internal Stresses in Nickel Wires.' *Zeitsch. für Metallkunde*, 1953, vol. 44, Apr., pp. 136–8.

Measurements of remanence, energy of magnetization and coercive force of a nickel wire are used for

calculation of the internal stresses present. The values obtained are compared with those given by X-ray methods of examination. Good agreement between the two sets of values was shown by the data for lightly worked material, but at higher degrees of elastic deformation the values for internal stress obtained by magnetic measurements were considerably below those resulting from X-ray determinations.

Production of Single Crystals of Nickel

R. F. PEARSON: 'Production of Single Crystals of Nickel.' *Brit. Jnl. Applied Physics*, 1953, vol. 4, Nov., pp. 342–4.

The aim of the paper is to show that large single crystals of nickel can be grown and orientated in a reasonably short time. A detailed account is given of production of the crystals by slow cooling from the melt, and of preparation of faces parallel to desired crystal planes. The necessity of melting the material in a vacuum is urged, and it is shown that this can be accomplished while the furnace windings are protected by an atmosphere of hydrogen. The particular problem with which the author was concerned was that of cutting a 'picture frame' from a single crystal. Methods of cutting and polishing crystal faces without introduction of undue strain are described.

Magnetic-Domain Studies on a Single Crystal of Nickel

L. F. BATES and G. W. WILSON: 'A Study of Bitter Figures on the (110) Plane of a Single Crystal of Nickel.' *Proc. Physical Soc. Section A*, 1953, vol. 66, Sept. 1, pp. 819–22.

Experiments were made by the powder-pattern technique, to determine the magnetic-domain structure of a single crystal of nickel cut so that two easy directions of magnetization lay in the plane of each main surface. The closure domain structure is a very simple one, and from the experimental evidence a model is proposed which is similar to that investigated theoretically by Néel in the case of a single crystal of iron.

Nitriding Characteristics of Nickel, Cobalt and Nickel-Cobalt Alloys in Relation to their Curie Temperatures

See abstract on p. 208.

Nickel-Alkaline Batteries in Searchlights

'Portable Battery Searchlight.' *Chemical Trade Jnl.*, 1953, vol. 133, Oct. 16, p. 970.

A new searchlight, now commercially available, is characterized by very high mechanical strength, and throws a long beam with wide angle diffusion. A 6/8-volt, 6-cell nickel-alkaline battery, having a nominal capacity of 11 Ah, powers a special long-life, clear-glass bulb with double bayonet contacts and twin filaments, taking, respectively, 3·0 and 0·5 amp. A full charge provides 3–4 hours' light on the main filament and about 20 hours on the reserve. Alternatively, a twin filament 1·0/1·0 amp. bulb can be fitted, to permit 9–10 hours' burning.

Structure of Nickel Tetracarbonyl

R. S. NYHOLM and L. N. SHORT: 'The Structure of Nickel Tetracarbonyl and Some Disubstituted Derivatives.' *Jnl. Chemical Soc.*, 1953, Sept., pp. 2670–3.

The structure of nickel carbonyl is discussed and it is shown that the infra-red spectrum and bond-length data are more compatible with a structure involving four Ni-C double bonds than with the generally accepted structure of two double bonds resonating among four positions. The stretching force constants of the C-O bonds in two disubstituted nickel carbonyls (*o*-phenylenebisdimethylarsine-dicarbonylnickel and dipyridyldicarbonylnickel) were calculated from the infra-red frequencies. Consideration of the data obtained leads to the conclusion that the metal-ligand bonds in these complexes possess considerable double-bond character.

Nickel Carbonyl: Toxicology Investigations

H. H. SCHRENK: 'Industrial Hygiene: Nickel Carbonyl.' *Industrial and Engineering Chemistry*, 1953, vol. 45, Oct., pp. 113A–14A, 116A.

Formation of nickel carbonyl, during the purging of a nickel-containing catalyst bed by an inert gas containing carbon monoxide, recently led to illness affecting a number of the staff employed in an oil refinery, thus emphasizing the necessity for adequate control measures in handling this gas. It is pointed out that in such a case danger can be avoided by employing nitrogen, but attention is drawn to the toxic character of nickel carbonyl in connexion with use which may be made of the gas in certain chemical syntheses.

The author summarizes results of some pertinent investigations of the effect, on mice, a cat, and rats, of exposure to nickel carbonyl, demonstrating the conditions which constituted (1) acute and (2) sub-acute, toxicity. Reference is also made to studies of the preventive effect of dimercaprol (BAL). To date, conclusions drawn with regard to the efficacy of BAL are conflicting, and it has been suggested that other thiols might be more effective than dimercaprol.

The information available indicates that response to exposure to nickel carbonyl is frequently delayed, and it is important that this fact should be borne in mind and that absence of immediate symptoms of poisoning should not be allowed to prevent prompt treatment after known exposure. It is noted that a threshold limit value of 1 p.p.m. for nickel carbonyl was accepted in 1952 by the American Conference of Governmental Industrial Hygienists, but the basis for selection of this value is not known to the writer of the present paper.

Nickel and Cobalt in Relation to Adherence of Enamel Ground Coats

See abstract on p. 206.

Plating by Thermal Decomposition of Volatile Metal Compounds

See abstract on p. 206.

Nickel-containing Temperature-Indicating Paints

J. F. COWLING, P. KING and A. L. ALEXANDER: 'Temperature-Indicating Paints.'
Industrial and Engineering Chemistry, 1953, vol. 45, Oct., pp. 2317-20.

The paper describes results of studies made in the Naval Research Laboratory, Washington, D.C.; the data are correlated with information available from other sources.

The authors conclude from their investigation that hundreds of compounds are of interest in connexion with temperature indication by colour. The distinctive properties applicable are characteristic of complexes of cobaltous chloride with ammonia, hydroxylamine, hexamethylene-tetramine, pyridine, ethylenediamine and hydrazine. Complexes of many additional cobalt salts are also possible, and analogies with compounds of iron, nickel and copper are likely to exist.

As an interim conclusion from the investigations of a large number of compounds, the authors list eleven cobaltous compounds and nickelous hexymethylene-tetramine bromide, as being of the greatest significance for practical applications. Temperature-sensitivity data are given for each of these complexes.

Use of Sodium Acetate in Estimation of Nickel

K. S. BHATKI and M. B. KABADI: 'Resacetophenone Oxime as a Reagent for the Quantitative Determination of Nickel.' *Jnl. Scientific and Industrial Research*, India, 1952, vol. 11B, p. 346.

Precipitation in the presence of sodium acetate is found to give quantitative separation of nickel as a bulky yellow-green complex. The accuracy of the estimation is stated to be comparable with that given by the conventional dimethylglyoxime procedure.

Spot Test for Copper, Cobalt and Nickel

L. A. GOEDBLOED: 'A Note on a Multiple Spot Test for Copper, Cobalt, and Nickel.' *Chemist Analyst*, 1953, vol. 42, June, p. 43.

SCHAEFFER recently described a spot test for copper, cobalt and nickel, based on the use of isoquinoline-ammonium thiocyanate reagent. (*ibid.*, 1952, vol. 41, pp. 57, 59; *Nickel Bulletin*, 1953, vol. 26, Nos. 8-9, p. 130). One limitation of the test is interference by excess ferric iron, which is a particular handicap in applying the method to ores.

To prevent such interference, the present author points out that ferric iron may be reduced with a solution of sodium sulphite. Ammonium thiocyanate in the reagent spot does not produce, with ferrous iron, a precipitate which would obscure the colours obtained from the cobalt and nickel. The copper in the cuprous state gives only a faint green colouration.

The recommended procedure is as follows: 'Heat a slightly acid solution of the sample to boiling. Add just enough of a 10 per cent. solution of sodium sulphite to effect reduction (which is complete when the yellow iron colour disappears), then add slight excess. Apply a drop of the reduced solution to filter paper treated by the Schaeffer method. In the presence of iron a thin red-brown line of ferric thiocyanate will develop (due to oxidation of some of the ferrous iron), but this in no way affects the clarity of the blue cobalt colour. On standing, the faint green colour due to copper will become more pronounced, due to oxidation of the cuprous ion.'

Determination of Cobalt in Nickel and Nickel Alloys

See abstract on p. 208.

Analysis and Determination of Metals and Alloys

See abstract on p. 202.

Analysis of Nickel Sulphate

J. KINNUNEN and B. WENNERSTRAND: 'Analysis of Nickel Sulphate.' *Chemist Analyst*, 1953, vol. 42, June, pp. 30-5.

Details are given of methods for complete analysis, covering determination of insoluble matter, nickel or nickel+cobalt, copper, iron, cobalt, zinc, lead. The procedures are based mainly on combinations of published methods: reference to the relevant literature is given. It is stated that the method is being successfully used in control of manufacture of commercial nickel sulphate to the following specification: nickel not less than 20·7, cobalt less than 0·1, copper less than 0·001, iron less than 0·001, zinc less than 0·005, lead less than 0·01, insoluble matter less than 0·005 per cent. With appropriate modifications, the same techniques can be applied to analysis of nickel-plating solutions.

ELECTRODEPOSITION AND OTHER COATING METHODS

Polishing of Metals for Plating: Influence on Quality of Coating

W. L. PINNER: 'A Further Study on the Effect of Abrasive Metal Polishing on the Character of Nickel Plate.' *Plating*, 1953, vol. 40, Oct., pp. 1115-18, 1123-5; disc., p. 1125.

This is the full form of the paper referred to, from an abstract source, in *Nickel Bulletin*, 1953, vol. 26, Nos. 8-9, p. 131.

The character of steel surfaces polished by various methods is illustrated by a series of photomicrographs, and the effect of the respective surface conditions, on the quality of superimposed coatings, is demonstrated by further photomicrographs and by critical discussion. A number of the examples shown relate to nickel coatings.

Consideration of the observations made leads the author to the following conclusions:—

Base-metal imperfections, such as pits and slag inclusions, are shown to have a most pronounced

effect on the corrosion-resistance of the plate. Such defects exert this influence in defiance of any type of polish used in the experiments described, or of any cleaning procedure. Either better steel, or the complete removal of such imperfections prior to the finishing operations, is preferred.

Fragmented metal particles produced in abrasive polishing are extremely damaging to plated coatings. Pre-finishing procedures should be designed to remove, as far as possible, slivers of steels and fragmented metal arising from the polishing operations. A final polishing with a lubricant is effective in partially accomplishing such removal.

An electrochemical treatment in which the steel is made anodic in suitable acid baths is more effective in removing slivers and fragmented metal. The one disadvantage of a sulphuric-acid anodic etch is the possibility of development of carbon smut on the surface of the steel, and this smut (due to too dilute a bath, too high a temperature or too low a current density) has been known to affect the buffability of dull or semi-bright nickel, and also the appearance of bright nickel coatings. Formation of smut can be avoided by rinsing the work after the anodic acid etch and following with an anodic alkaline electrolytic cleaning treatment. The steel is finally rinsed and then treated in a mild muriatic-acid solution. Surfaces so treated possess all the characteristics conferred by the anodic acid-etch, without risk of impairment of surface.

Nickel Plating of Brass Tags in Post Office Equipment

H. F. HOURIGAN, J. W. ROBINSON and W. T. EDWARDS: 'Poor Solderability of Tags and Dry Joints.' *Post Office Electrical Engineers' Jnl.*, 1953, vol. 46, Oct., pp. 134–9.

Many of the brass connexion tags used in Post Office equipment are finished by mass dipping in a hot solder bath, such solder coat being utilized both as a protective finish and as a preparation for soldering the tags onto the apparatus. Tags so processed had been found to exhibit widely varying degrees of solderability. This paper reports an investigation of the cause of the variation, and of methods devised for rendering the tags consistently solderable.

Investigation of commercially treated samples, combined with laboratory tests (details of which are given) showed that, in mass soldering, the copper and zinc constituents of the brass dissolve into the hot solder and contaminate the bath, with the result that the surface of many of the tags has a high zinc content, with associated bad solderability.

Nickel plating of the tags before coating has been found effectively to prevent contamination of the solder bath and of the coating produced from it, providing that the nickel coating is not less than 0·0001 in. thick and that in solder-coating the temperature of the bath is not above 250°C. The preferred temperature of solder coating is about 200°C.

In view of the fact that any nickel coating will be dissolved to some extent by molten solder, tests were made to determine the effect of nickel-contamination on corrosion-resistance of solder. Exposure comprising steam-ageing, for periods of up to 500 hours, of solders containing 0·018 per cent. of nickel (the nickel content of a solder bath which had been in contact with nickel for 8 hours) showed that such slight contamination has no injurious effect on the solder. The rate of corrosion in the conditions used was very similar to that of pure solder.

Plating of Molybdenum with Nickel and Other Metals

A. KORBELAK: 'Plating on Molybdenum.' *Plating*, 1953, vol. 40, Oct., pp. 1126–31; disc., pp. 1131–3.

Attempts to use molybdenum in elevated-temperature applications have shown that the metal is unstable in air at temperatures above about 700°C. In order that advantage may be taken of the combination of high strength, hardness and abundant availability of molybdenum, it is therefore highly desirable to devise methods for coating it with other metals which have greater resistance to oxidation.

The author of this paper summarizes the methods which have been proposed for production of coatings on molybdenum:—

(1) *Pack Method.* Molybdenum parts are packed in powdered chromium, nickel and other metals, and heat is applied. Large and specialized equipment is required, and unusual shapes are difficult to accommodate.

(2) *Reduced-Oxide Method.* Nickel oxide in powdered form, dispersed in a suitable vehicle such as lacquer, has been used with some success in certain applications. The coating is brushed or sprayed on, or the article is dipped, and the coated material is heat-treated in a furnace having a reducing atmosphere. The vehicle is burned off, leaving the reduced-oxide film. As with the pack method, the need for special equipment, and limited size of furnaces, preclude wide use of this procedure.

(3) *Gas-Plating Methods.* Chromizing techniques have also been employed, involving the use of the vapour of a metal halide generated by the action of hydrochloric acid and hydrogen on chromium particles in contact with molybdenum. Deposition of metal coatings by thermal decomposition of volatile metallic compounds, e.g., carbonyls, is covered by a number of recent patents, and the process is finding some practical application: see abstract on p. 206.

(4) *Diffusion Method.* Powdered metal, e.g., nickel, in a vehicle such as amyl acetate, is applied, and the coated material is heat-treated to destroy the vehicle. The coating so produced is undesirably rough, and the specialized equipment required is a further objection to the process. In a variant of this method, a layer of nickel is electrodeposited and the coated material is heat-treated, but it has been found difficult to obtain satisfactory adhesion between base and coating.

The experimental work reported by the present author was designed to develop an improved electroplating technique. During electrodeposition, onto molybdenum, of an 80–20 nickel-chromium alloy (by deposition of alternate layers of the two metals and subsequent heat-treatment in chromium powder), it was observed that chromium does not alloy with molybdenum except when in direct contact with aluminium oxide, and that chromium plating on properly cleaned molybdenum gives good adherence. This finding led to the development of the chromium-strike barrier technique which is the basis of the method now proposed.

The cycle recommended comprises the following steps: Anodic cleaning, in sulphuric-acid solution; water rinsing; alkaline rinsing; water rinsing; rinsing in 10 per cent. sulphuric acid; water rinsing; chromium striking, in a standard chromium-plating bath, operated at a temperature not below 120°F. (50°C); water rinsing; nickel striking from a chloride bath, preferably worked at a current density of about 50 amp./sq. ft.; rinsing; final plating with nickel or any other metal which is required as the outer coating. It has been found that this procedure is also satisfactory for plating of steels containing molybdenum.

Some useful applications of plated molybdenum include resistance welding tips, parts of electronic equipment, and electrodes of glass furnaces.

Bright Nickel Plating: The 'Nickelume' Process

'Plating: Develop Better Process.' *Chemical Engineering News*, 1953, vol. 31, Oct. 19, pp. 4310, 4313; see also *Iron Age*, 1953, vol. 172, Oct. 1, p. 44.

The following claims are made, by Hanson-Van Winkle-Munning Company, for a new organic-type nickel-plating process which is being publicized under the name of 'Nickelume':

The bath produces bright deposits, of pleasing colour; good levelling and smoothing is obtained; a wide range of operating temperature is permissible, without loss of brightness; the deposits show good resistance to corrosion; a wide range of current density is permissible; the deposits are ductile and show only low compressive or tensile internal stress; the bath has good tolerance for impurities; no activation of the nickel coatings is required prior to plating with chromium or other metal; composition of the bath is easily controlled, and the bath is stable over long periods of operation.

Some particulars are given of preparation of the bath, and of temperature and other operating conditions. It is claimed that the coating shows a degree of brightness which suggests that in some cases final chromium plating could be eliminated.

Electrodeposition of Metal Coatings by a Brushing Method

'The Dalic Plating Process.' *Machinery* (*Lond.*), 1953, vol. 83, Oct. 23, pp. 824–7.

An account is given of a process (originally developed in France, and now available in the United Kingdom) according to which metallic coatings are deposited by brushing the electrolyte onto the basis-metal surface, using an absorbent pad attached to a tool or 'tampon', which is made the anode of an electrical circuit, with the workpiece forming the cathode. Equipment, solutions, and technique of operation are described, with reference to electrodeposition of nickel and a number of other metals. Some applications of the process are also mentioned.

Plating by Thermal Decomposition of Volatile Metal Compounds

'Gas Plating Offers Versatility.' *Steel*, 1953, vol. 133, Oct. 19, pp. 120–1, 124.

Plating of metals by thermal decomposition of metal carbonyls, a logical outcome of the Mond carbonyl nickel-extraction process, is being exploited in large-scale operation under patents held by Commonwealth Engineering Company, Dayton, Ohio, on whose experience and methods this article is based. The essential feature of the process is the circulation of the carbonyls (or other suitable gaseous metal compounds) round the object to be plated, which is heated to a temperature at which the gas decomposes, with resultant plating out of metal. The method is illustrated by a diagram showing the equipment and cycle of operations used for continuous plating of strip. Wire screen, paper, and ceramics can also be plated by the gas method.

The article includes data on the characteristics of the carbonyls of nickel, iron, chromium, molybdenum, tungsten, cobalt and ruthenium, and lists some applications in which gas plating has proved successful. These include heavy nickel plating on lead tyre moulds; thin coatings on tubes and pipes, wire, and aluminium discs for electrical purposes; thin coatings on magnesium and on cadmium; coatings of various thicknesses on strip steel; coatings on sintered brass; on tiles (for decorative purposes); on glass fibres (for imparting electrical conductivity) and on Teflon (for electrical uses). Applications which have been developed for gas-plated coatings of copper, chromium, iron, tungsten, and silver are also listed.

It is pointed out that, as a supplement to electrodeposition, the gas-plating procedure has several advantages, among which are the following:—non-conductors, such as paper, plastic and ceramics, can be plated; irregular shapes can be treated without special equipment; the method is very rapid, and there is no risk of hydrogen embrittlement.

Nickel and Cobalt in Relation to Adherence of Enamel Ground Coats

R. F. PATRICK, E. G. PORST and G. H. SPENCER-STRONG: 'Study of Some Phenomena Associated with the Adherence of Sheet Iron Ground Coats.' *Jnl. Amer. Ceramic Soc.*, 1953, vol. 36, Sept. 1, pp. 305–13.

The authors point out that, in spite of the large amount of data which has been accumulated over many years, no single theory explaining the mechanism of enamel adherence has yet been universally accepted. The main features of four theories which

have been advanced by various writers are briefly summarized under the headings (1) mechanical theory, (2) dendritic theory, (3) oxide-layer theory, and (4) electrolytic theory.

The work described in this paper originated in observations made by the authors, some years previously, on de-enamelling of ground-coated plates with a view to determining the amount of nickel deposited during nickel dipping. In every case it was found that the nickel deposit was larger than was expected, and a further finding was that some plates which had not been nickel-dipped showed nickel on the surface after de-enamelling. Later experiments established the fact that cobalt also was deposited on the metal surface during firing of sheet-iron ground coats. In view of these results, a research programme was instituted to study the phenomena observed and to correlate them with the adherence behaviour of the enamels under study. The present paper reports results of part of this investigation.

Most of the ground coats studied were either commercial enamels or variations of commercial types. The experimental work was divided into four main sections:—

(1) describes deposition of cobalt and nickel on the surface of the metal as a function of temperature and composition of the enamel.
(2) reports experiments made to determine the nature of these coatings.
(3) deals with relationship between deposition of cobalt and of nickel and solution of iron in the ground coat.
(4) concerns relation between the amount of cobalt or nickel deposited and adherence of the enamels.

A large amount of experimental data is recorded, and the results of the individual series of tests are critically discussed. The following general conclusions are drawn:—

'Cobalt and nickel deposit on the surface of the base metal during firing of sheet iron ground coats.

'These deposits, which are metallic in nature, at least at higher firing temperatures, increase with increasing firing temperature. They also increase with the amount of cobalt or nickel present in any one frit and with the fluidity of the enamel. One reason why cobalt is generally considered to be a better adherence agent than nickel is that cobalt has a greater tendency to deposit during firing. Ground-coat adherence is generally associated with a certain amount of deposition of cobalt and nickel on the metal surface. This deposition is in turn associated with a roughening of the surface of the metal, which is believed to be a large contributory factor in enamel adherence.'

Thickness Testing of Organic Bright-Nickel Deposits

'The B.N.F. Jet Test on Organic Bright Nickel Deposits.' *Electroplating*, 1953, vol. 6, Nov., pp. 417–18.

The B.N.F. Jet Test, developed by S. G. CLARKE, has been for many years widely used in England for determination of thickness of electrodeposited coatings of nickel, but it is recognized that results obtained by it on organic-type bright-nickel coatings are not strictly comparable with measurements made on other nickel coatings. A working factor which has been used to compensate for this difference is accurate only within a fairly narrow range of thickness of deposit, and rate of penetration of the coating may vary considerably on deposits made from different organic solutions.

The present note is based on work in the laboratories of the British Non-Ferrous Metals Research Association, as a result of which calibration curves have been developed making possible estimation of true thickness of deposit from an apparent thickness given by the standard procedure used in the test, applying the normal temperature correction. Curves relating to deposits made from Efco-Udylite and from Super Gleamax solutions are shown. The work done in this connexion also established that the normal temperature-dependence of the jet test is not significantly changed in testing bright nickel deposits, and that while annealing at 400°C. reduces rate of penetration, such treatment does not significantly affect the variation of the latter with thickness of deposit and type of brightener.

Evaluation of Porosity of Metal Coatings by Analysis of Corrosion Products

N. THON, L. YANG and S. YANG: 'Porosity of Electrodeposited Metals. XI. Analysis of Corrosion Products as a Method of Measuring the Change of Porosity in Corrosion.' *Plating*, 1953, vol. 40, Sept., pp.1011–15.

The report is one of the series issued by A.E.S. Research Project 6. It describes a method for analysis of corrosion products formed on plated steel, as a means of determining progress of porosity. The problem of separation of the corrosion products from the metal, in such a way as to ensure collection of all such products without intensifying the corrosion of the base, was solved by rubbing the surface with a rubber-tipped glass rod (a 'policeman', such as is used in chemical analysis), followed by a chemical treatment. By this method the major part, and in some cases all, of the oxidized iron was removed, and was collected in water which was then acidified to dissolve the oxides. After further treatment, full details of which are given, the solution was analyzed for iron, nickel, copper. Examples are given to demonstrate the application of the method to specimens subjected to various corrosive exposures, including immersion in hot water (with and without access of air), intermittent immersion in salt solution, and exposure to a drastically corrosive gaseous atmosphere (over a saturated solution of sulphur dioxide in water).

Measurement of Surface Roughness of Electrodeposited Coatings

N. THON, L. YANG AND S. YANG: 'Porosity of Electrodeposited Metals. XII. Measurement of Surface Roughness and its Change in Exposure.' *Plating*, 1953, vol. 40, Oct., pp. 1135–7.

Report of work done under A.E.S. Research Project No. 6.

Earlier research under this Project has led to the recognition of two types of corrosive attack, surface

and depth corrosion. The latter occurs at discrete points, broadening existing fine pores or producing further porosity, and finally causing failure where the base metal is laid bare and is directly accessible to the corrosive agent. Surface corrosion leads to failure only indirectly, insofar as it causes the protecting metal coating to become progressively thinner and thus hastens the progress of depth corrosion.

The effectiveness of surface corrosion in accelerating depth corrosion and early occurrence of local failures depends on the extent to which the attack is non-uniform. There is therefore obvious need for means of evaluating the distribution of surface corrosion from the point of view of its uniformity. The method of assessment proposed in this paper is based on the relation between the apparent surface area and the true surface area of a chosen portion of an electrodeposited coating. The ratio of real to apparent surface area is the roughness factor, R, and the change of this value, over a period of exposure to a selected corrodent, is measured as a gauge of surface corrosion. An example relates to a Watts-type nickel deposit exposed to hydrochloric-acid vapour.

Porosity Testing of Coatings on Zinc-base Materials

M. EITEL: 'Tentative Specification D.I.N. 50956 for Testing of Porosity in Coatings on Zinc and Zinc Alloys.' *Werkstoffe und Korrosion*, 1953, vol. 4, No. 10, pp. 356–7.

This proposed standard, dated August 1953*, is intended for testing porosity in coatings which are more noble than the zinc or zinc-alloy base: it is specially suitable for use on coatings of copper+nickel and copper+nickel+chromium, and is designed to reveal pores which penetrate through to the basis metal. It does *not* constitute a test of the presence of cracks present only in the thin outer coating of chromium.

The solution used is made by dissolving 5 g. of copper sulphate ($CuSO_4.5H_2O$) in 95 ml. of water. In order to neutralize free acid, a small quantity of basic copper carbonate is added. The neutralized solution should be filtered after it has settled.

The sample to be tested is immersed for three minutes in the above solution, and dried in air. Presence of pores is revealed by reddish copper spots which are usually visible to the naked eye. Large pores show up as dark-grey spots surrounded by a copper-coloured ring. The porosity is reported as number of pores per square decimetre.

NON-FERROUS ALLOYS

Spot Test Differentiation between Monel and Nickel Silver

C. GOLDBERG: 'Spot Test Differentiation of Monel Metal and Nickel Silvers.' *Chemist Analyst*, 1953, vol. 42, June, p. 35.

The author has earlier described a rapid method for distinguishing between Monel and nickel silvers (*ibid.*, 1950, vol. 39, pp. 56–7; 1951, vol. 40, p. 38; *Nickel Bulletin*, 1950, vol. 23, No. 12, p. 225; 1951, vol. 24, Nos. 9–10, p. 194). The procedure recommended requires colorimetric equipment for measurement of the colour of nickel chloride.

The present note gives details of a qualitative method which may be used by laboratories in which such equipment is not available.

'Weigh and place $0{\cdot}10 \pm 0{\cdot}01$ g. of the metal in a 500-ml. volumetric flask and add 10 ml. of nitric acid (concentrated, diluted 1:1). Warm until solution is complete and dilute to mark with distilled water. Mix well and transfer one drop to a white spot plate. Add one drop of aqueous ammonia and one drop of a 3 per cent. aqueous solution of sodium dimethylglyoximate. Nickel silvers give a pale brown coloration, with no precipitate, within five minutes. Monel gives a bright red coloration and immediately gives a definite precipitate. There is no need for a complexing agent for elements which might be precipitated by ammonia, since they do not interfere at the dilution specified.'

Determination of Cobalt in Nickel and Nickel Alloys

M. JEAN: 'Colorimetric Determination of Cobalt as Tetraphenylarsonium Cobaltothiocyanate, in Nickel and Copper-Nickel-base Alloys.' *Revue du Nickel*, 1953, vol. 19, July-Sept., pp. 63–5.

In 1951 AFFSPRUNG *et al.* described the spectrophotometric determination of cobalt as tetraphenylarsonium cobaltothiocyanate, applying the procedure to alloy steels, and nickel-copper alloys, as well as to other materials. (See *Nickel Bulletin*, 1952, vol. 25, No. 2, p. 52.) The present author has studied the application of the method for determination of cobalt in nickel, copper-nickel alloys, and nickel silvers. The work amplifies that of the earlier investigators, and directs attention to the interfering influence of zinc, which precludes the use of this procedure for estimation of cobalt in nickel silvers.

Radiation Properties of Nickel-containing Materials at Elevated Temperatures

See abstract on p. 212.

Nitriding Characteristics of Nickel, Cobalt and Nickel-Cobalt Alloys in Relation to their Curie Temperatures

G. NURY: 'Study of the Nitriding of Some Nickel-Cobalt Alloys in the Region of their Curie Points.' *Comptes Rendus de l'Académie des Sciences*, 1953, vol. 237, Sept. 28, pp. 654–6.

Brief report of experiments on nickel, cobalt, and four binary alloys of these metals, containing, respectively, 12, 10, 9, and 8 per cent. of cobalt. The Curie points of these alloys are in the range 440°–540°C.

Nitriding treatment was applied (at temperatures of 300°–600°C.), and its effect on hardness and structure of the surface of the materials was determined. The influence of the magnetic transformation was demonstrated by a sharp maximum in rate of nitriding at

*Comments received up to January 31, 1954.

the Curie-point temperatures of the respective materials. The author refers to earlier work of a similar character, on iron (*ibid.*, 1953, vol. 236, p. 1487).

CAST IRON

Transformation Phenomena in Alloy Cast Irons

J. VAN EEGHEM, J. VIDTS and A. DE SY: 'Isothermal Transformations and the Heat-Treatment of Copper-Chromium and Nickel-Chromium Cast Irons.' *International Foundry Congress*, 1953, Sept., Paper CI-17; 28 pp.

The authors report results of a study of the isothermal transformations in some alloy cast irons, using metallographic examination and a dilatometric method developed in the laboratories of the University of Ghent, in collaboration with Le Centre d'Etude et de Recherches en Fonderie. The determinations made have established TTT diagrams for four types of cast iron, the compositions of which are shown below:—

C %	Si %	Mn %	S %	P %	Cr %	Cu %	Ni %
3·08	1·80	0·83	0·07	0·09	0·23	1·02	—
3·04	1·79	0·85	0·07	0·08	0·24	1·97	—
3·00	2·04	0·81	0·07	0·08	0·26	—	0·99
3·15	2·09	0·84	0·08	0·09	0·25	—	2·04

Following a detailed investigation of the isothermal transformations occurring in the upper and lower bainite regions and in the pearlite region, the authors studied the influence of copper and of nickel on these transformations. Some of the findings with regard to isothermal transformations supplement those which have been established by the study of transformations in steels.

In conclusion, attention is directed to the possibility of isothermal and 'stepped' treatment of cast irons, and the practical utility of such treatments as a means of securing certain desired combinations of properties.

Arc Welding of S.G. Iron

T. E. KIHLGREN and H. C. WAUGH: 'Joining of Ductile Iron by Several Arc Welding Methods.' *Welding Jnl.*, 1953, vol. 32, Oct., pp. 947–56.

With the increasingly wide acceptance of spheroidal-graphite cast iron, in many fields of application, weldability is becoming an important consideration, and the need for more information on the response of the material to welding, in fabrication and salvage, is obvious. This paper reports work done to establish practical data on metal-arc, inert-gas-shielded, and submerged-arc welding of magnesium-treated cast iron, using 60–40 nickel-iron electrodes and filler wires. Some information is also given on metal-arc welding with steel electrodes.

On the basis of the detailed results which are reported, the authors consider the following conclusions to be justified:

S.G. iron can be welded by most of the commonly used fusion processes.

To obtain best response to welding, the iron should be used in the fully annealed condition, but, by suitable selection of composition of casting and procedure, it is possible to weld as-cast material.

The lower the content of alloying elements which favour hardenability, the better is the response of the iron to welding and the less essential is pre-heating. Phosphorus has an adverse influence on weldability: 0·10 per cent. of this element is the maximum permissible when welding with nickel-iron electrodes or filler wire, and if mild-steel electrodes are employed the phosphorus should not exceed 0·05 per cent.

Iron of the following composition was found to respond satisfactorily to welding by several processes: total carbon 3·60, silicon 2·40, manganese 0·30, nickel 0·70, phosphorus less than 0·05, magnesium 0·05–0·08, per cent.

Metal-arc welding, using austenitic nickel-iron electrodes, and with and without pre-heat, was successfully applied to castings over a fairly broad range of composition, in both the as-cast and the annealed conditions. Inert-arc welding with the nickel-iron filler wire shows promise of successful development: joints in S.G. iron of preferred composition, welded in both the as-cast and the annealed conditions, were readily made by this method in plate up to 1 in. in thickness.

S.G. iron may be joined, by means of nickel-iron electrodes, to dissimilar metals such as mild steel, stainless steel, nickel, Monel, and Inconel. This possibility is of particular practical value in the many field applications in which S.G. iron/mild steel joints are required.

Tests on welds in various conditions (details of which are given) show that in applications in which machinability is a primary requirement arc-welded joints should be fully annealed (treatment at 1650°F. (900°C.) followed by heating at 1275°F. (690°C.)). In some cases the 1275°F. treatment alone may be sufficient. A considerable degree of improvement in machinability of as-welded joints can be effected by means of special welding techniques, and by brief torch-annealing treatments, but the machining qualities of joints so treated will not be equal to those of fully annealed welds.

Where a hard heat-affected zone exists it was found that tungsten-carbide-tipped tools were to be preferred to tool-steel bits. Low-hydrogen mild-steel electrodes offer some promise for joining S.G. iron, provided that (*a*) the castings are low in alloy content, (*b*) phosphorus is kept low, and (*c*) the castings are welded in the fully annealed condition.

Experiments with the submerged-arc process were confined to joining of S.G. iron to mild steel. On the basis of these limited experiments, the method appears likely to be useful for some special applications, but its field is likely to be limited.

It is emphasized that the studies reported in the paper must be regarded as only an introduction to full investigation of the advantages and limitations of the various arc-welding processes, and of the compositions of S.G. iron which will give optimum response to welding. It is recorded that, although the authors make no reference to oxy-acetylene welding of S.G. iron, this method is being successfully used in some foundries, employing S.G. iron filler rods.

CONSTRUCTIONAL STEELS

Spectrographic Analysis of Low-Alloy Steels

BRIT. STANDARDS INSTN.: 'British Standard Recommended Method for the Spectrographic Analysis of Low-Alloy Steels.' *B.S. 1121B*: 1953. Price 5/-.

The schedule has been prepared, under the authority of the Iron and Steel Institute, as part of a programme of standardizing methods for the determination of elements in iron and steel.*The recommendations made are based on the work of the Spectrographic Analysis Sub-Committee of the British Iron and Steel Research Association. The introductory section of the specification includes general notes on the method, form of sample, etc., and gives the following list of the elements and concentrations covered by the recommended method, viz.:—

Element	%
Silicon	0·05–0·80
Manganese	0·05–1·50
Nickel	0·10–5·00
Chromium	0·05–3·00
Molybdenum	0·05–1·50
Vanadium	0·03–0·65
Copper	0·05–0·50

Section 2 gives details of apparatus required, and of standard steels which may be used for calibration of equipment and other comparisons; Section 3 describes analytical procedure; Section 4 gives methods for plate calibration and standardization; Section 5 contains a short note on reproducibility. These details are supplemented by a reference table summarizing essential features of analytical procedure, a table showing recommended line pairs, a table of standard iron lines, and a figure giving typical low-alloy-steel spectra.

Photometric Determination of Titanium in Iron and Steel

See abstract on p. 216.

HEAT- AND CORROSION-RESISTING MATERIALS

Properties of Alloys of the Nimonic Series

HENRY WIGGIN AND CO., LTD.: 'Supplementary Data on the Nimonic Alloys.' *Publn.* 688*,1953; 8 pp.

This publication is issued as a supplement to the data book entitled 'The Nimonic Alloys' (No. 459A), in order to make available, as rapidly as possible, additional information which has been established since the issue of the last edition of the main publication.

The scope of the contents of the supplementary issue is shown below: the data are in each case presented in tabular form:—

- Physical Properties of Nimonic D.S.
- Fatigue Properties of Nimonic 80A and 90.
- Torsion Properties of Nimonic 80A and 90.
- Short–Time Creep–Test Data on Nimonic 95 Hot–Rolled Bar.
- Recommended Heat–Treatments for Nimonics 75, F, 80, 80A, 90 and 95.
- Acceptance Creep Tests laid down by D.T.D. Specifications for Nimonic 80, 80A, 90, and specification requirements for Nimonic 95 to be embodied in a D.T.D. specification not yet issued.

In each case the relation of the supplementary data to information already given in Publication 459A is indicated. The amendments will be embodied in the next revision of the main publication.

Nimonic Alloys in Aircraft Gas Turbines

HENRY WIGGIN AND CO., LTD.: 'Nimonic Alloys in Aircraft Gas Turbines.' *Publn.* 686, 1953; 11 pp.

The spectacular development of the gas turbine as a power unit for aircraft propulsion has been made possible only by production of materials which will withstand the severe operating conditions. Although the principles of operation of the gas turbine had been known for many years, it was not until the war period that alloys were developed which would give satisfactory service in such units, particularly in the conditions of high temperature and high stress in which the moving blades operate. Materials meeting these requirements have been developed under the name of the Nimonic series of alloys, and to date every British aircraft gas turbine which has gone into

* See *Nickel Bulletin*, 1953, vol. 26, No. 4, p. 57.

* *We shall be pleascd to supply a free copy of this publication.*

production has been equipped with moving blades made from one or other of the alloys of this series.

This short brochure gives some notes on research, in the laboratories of The Mond Nickel Company, Ltd., which led to development of the alloys, and of the closely controlled operations by which they are commercially produced in the works of Messrs. Henry Wiggin & Company, Ltd.

The centre pages contain a diagram showing the gas flow of the Rolls-Royce Derwent 5 turbo-jet engine, selected as typical of a modern gas turbine. Temperatures reached in the various parts of the engine, under operating conditions, are illustrated by colour variations.

The remainder of the publication comprises condensed information on the properties of six of the Nimonic alloys, a list of the complete range of Nimonic alloys now available, and notes on some typical applications of these materials in aircraft.

Formation of Sigma Phase in 25-12 and 25-20 Chromium-Nickel Steels

R. C. FRERICHS and C. L. CLARK: 'The Formation of Sigma Phase in Types 309 and 310 Stainless Steel.' *Amer. Soc. Metals, Preprint 9W*, 1953; 12 pp.

In the 'Resumé of High-Temperature Investigations conducted during 1948-50', issued by the Timken Roller Bearing Company, reference is made to the presence, in an austenitic steel, of a 'dark needle-like constituent' which was tentatively identified as a carbide. The suggestion was made that this phase might possibly be an early stage in the formation of sigma. The purpose of the studies reported in the present paper was (*a*) to determine whether any relationship existed between the 'dark needle-like constituent' and sigma, and (*b*) to investigate the formation of sigma as affected by grain size, time of heating, and temperature.

The heats used in the experiments were of the compositions shown below:—

Type	C %	Mn %	P %	S %	Si %	Cr %	Ni %	Mo %	N_2 %
309	0·088	1·44	0·020	0·012	0·58	23·66	13·82	0·03	0·132
310	0·107	1·55	0·020	0·013	0·39	25·05	20·80	0·03	0·136

Two basic structures, resulting from different prior treatments, were used: (*a*) to produce a fine-grained structure specimens of each steel were air-cooled from 1800°F. (980°C.), after being cold-finished from the hot mill, and (*b*) the coarse-grained structure was obtained by water-quenching from 2150°F. (1175°C.). Both fine- and coarse-grained specimens were aged in a gradient furnace, for 100, 500 and 1,000 hours. With the furnace used it was possible to produce a uniform and reproducible gradient of about 600°F. in any pre-determined temperature range: for these experiments the range selected was 1050°-1650°F. (565°-900°C.).

After completion of the ageing treatments, a hardness survey was made over the full length of the specimens, after which Izod impact test pieces were machined from the bars. Metallographic examination, magnetic-permeability measurements and X-ray analysis of the treated material were also made. The paper contains a large number of photomicrographs illustrating the structure of the steels in the various conditions.

Correlation of the results obtained by the various methods of examination leads to the conclusion that sigma formation is more rapid in the fine-grained structure (as produced by cold work followed by normalizing at 1800°F.: 980°C.) than in the coarse-grained water-quenched steels. There is also a greater tendency for formation of the needle-like constituent in the coarse-grained materials. Both phases form in the same general temperature range, and both tend to lower the room-temperature impact-resistance. The sigma has the greater effect. The author emphasizes, however, that such conclusions do not imply that the coarse-grained structures are superior for high-temperature applications: the reverse is true if appreciable ductility to fracture is essential.

The steel containing only 12 per cent. of nickel showed a greater tendency towards formation of the needle-like constituent than did the higher-nickel (20%) steel, a difference which is probably attributable to the greater stability of the austenite in the higher-nickel steel.

Work-Softening of Austenitic Chromium-Nickel-Molybdenum Steel at Elevated Temperatures

D. A. J. MILLER and J. WULFF: 'Work-Softening of 16-25-6 Alloy at Elevated Temperatures.' *Amer. Soc. Metals, Preprint 12W*, 1953; 10 pp.

An important characteristic of many engineering materials is their capacity to harden under strain, and the present authors (in an investigation not yet reported) have shown that steel strain-hardens, even at elevated temperatures, provided that the rate of strain exceeds the rate of recovery and recrystallization.

The studies reported in the present paper have, however, established that a steel containing carbon 0·1, chromium 16, nickel 25, molybdenum 6, per cent. can be *softened* by plastic working at elevated temperatures, and that by suitable ageing after working the mechanical properties of the material can be markedly improved. The test used to evaluate the changes producible by such treatment was the short-time tensile test, at constant rate of strain. The strain rates employed varied from 9×10^{-5} to 600×10^{-5} per second, and covered a temperature range of 1800°-2200°F. (980°-1205°C.).

In explanation of the softening observed in this material with increasing strain, it is postulated that the deformation causes solution of a portion of a grain-boundary precipitate present in the steel, so softening the material, and that re-precipitation along the planes of deformation on ageing gives increased strength. The validity of the assumption was demonstrated by

results of metallographic examination. Of four possible explanations for the solution effect induced by strain, the most feasible is considered to be changes in the equilibrium state due to internal local stresses.

Properties Required in Creep-resisting Steels

E. W. COLBECK, J. R. RAIT and J. O. WARD: 'The Design of Creep-Resisting Steels.' *Engineering*, 1953, vol. 176, Oct. 16 and 23, pp. 505–6, 537–40. (Paper read before Section G of the British Association, September, 1953.)

After a brief reference to some of the early literature of creep, the authors enumerate the properties essential in high-temperature materials, emphasizing the problems involved in obtaining the desired combination of properties. Criteria used for assessment of creep-resistance at high temperature are discussed, and typical creep-testing equipment is described by reference to machines in the laboratories of Messrs. Hadfields, Ltd.

The second section of the paper reviews the types of material which have been developed for high-temperature service; representative compositions are quoted and typical creep properties for each compositional group are tabulated. Some reference is made to specific investigations which have resulted in evolution of various steels and alloys, and the influence of individual elements and of heat-treatment is discussed. The essential features of composition, for austenitic and ferritic high-temperature materials, are summarized, and some forecast is made of probable lines of future research in this field.

Production and Properties of Bolts for Use at High Temperatures

T. W. HARKER: 'Manufacture and Characteristics of High-Temperature Bolts.' *Metal Progress*, 1953, vol. 64, Oct., pp. 125–8.

Design of high-temperature bolting requires consideration of many factors, including resistance to oxidation and corrosion, creep-resistance, fatigue strength, notch-sensitivity and stress-rupture properties. Time-temperature-stress relationship is obviously the most difficult feature, in view of the complications introduced by elevated temperatures and corrosive environments. The use of the alloys developed to meet these essential requirements has necessitated specialized methods of fabrication, and this article, describing practice in the aeronautical division of a large American firm, gives an account of some of the procedures now used.

Basically, there are two methods of producing bolts: (1) the blanks may be machined from a hexagonal bar which has the same hexagon size as the bolt head; or (2) the bolt head is upset from a round bar of approximately the same diameter as the shank. Threads may be either cut or rolled, but for most high-temperature applications rolled threads are required by many specifications.

Most of the materials used for high-temperature bolting are of the high-alloy austenitic types, and for various reasons, which are detailed in the text, machining from hexagonal bar is not an economic method of fabrication. A process involving cold forging and hot upsetting is preferred. The author comments on the close control of temperature and working conditions required to give optimum properties in the finished bolts, and refers to heat-treatments suitable for age-hardenable and non-age-hardenable materials.

The following materials are listed as being relevant to the discussion of high-temperature bolting: 18–8 chromium–nickel steels of straight, stabilized, molybdenum-containing, and age-hardening types; straight-chromium steels and high-chromium steel of free-machining grade; Inconel 'X' (precipitation-hardening nickel–chromium-base alloy); 16–25–6 chromium–nickel–molybdenum steel; 19–9–DL and Discaloy (typical of the iron-base complex alloys which have been developed for various forms of high-temperature service).

Radiation Properties of Nickel-containing Materials at Elevated Temperatures

J. POGGIE: 'Radiation Characteristics of Metals.' *Product Engineering*, 1953, vol. 24, Sept., pp. 205, 207.

Series of charts showing the relative effects of surface condition and composition on the radiation characteristics, at elevated temperatures, of the materials listed below. The data are expressed as per cent. emissivity, which equals—

$$\frac{\text{radiation intensity of specimen}}{\text{radiation intensity of a black body}}$$

The following comments are made on the information so presented:—

Reflection characteristics in the infra-red region are modified by oxidation and surface condition. Colour appears to have no detectable effect.

Alclad. Polishing has little or no effect, due probably to the high polish usually present on stock of this material. Prolonged exposure at 400°F. (204°C.) resulted in marked increase in emissivity of both polished and unpolished specimens.

Titanium. Emissivities of polished and unpolished (oxidized) titanium were identical. After 48 hours at 800°F. (426°C.) emissivity of the unpolished surface was 5–12 per cent. higher than that of the polished one.

Stainless Steel (composition not given). Polishing caused substantial decrease in emissivity. Prolonged exposure at 800°F. (426°C.) had no effect, but emission increased when the specimens were oxidized at 1200°F. (648°C.).

Nickel Alloys (*Inconel, Inconel X, and Monel*). Both polished and unpolished specimens of all three alloys exhibited marked increase in emission after exposure at 1200°F. (648°C.) for 48 hours.

Corrosion of High-Temperature Materials by Vanadium Pentoxide

S. H. FREDERICK and T. F. EDEN: 'Corrosion Aspects of the Vanadium Problem in Gas Turbines.' *Instn. Mechanical Engineers, Advance Copy*, 1953; 8 pp.

This paper reports results of laboratory investigations, made in the Pametrada Research Station, to

determine the behaviour of the following materials when in contact with vanadium pentoxide and mixtures of vanadium pentoxide and sodium sulphate.

I. High-Temperature Materials

Steel F.C.B. (T) (carbon 0·12, manganese 1·50, nickel 11·0, chromium 17·5, niobium 1·2, per cent.).

Steel G.18B (carbon 0·40, manganese 0·80, nickel 13·0, chromium 13·0, molybdenum 2·0, tungsten 2·5, niobium 3·0, per cent.).

Nimonic 80 (carbon 0·09, manganese 0·30, chromium 20, aluminium 0·5, titanium 2·5, iron 0·4, per cent., nickel balance).

This group of materials was tested in—

V_2O_5 (670°C.) .. .. .. } For various periods

V_2O_5 + 10% Na_2SO_4 (670° and 820°C.) } For various periods

V_2O_5 + 30% Na_2SO_4 (670°C.) .. } For various periods

II. Nickel and Chromium Coatings Electrodeposited on Mild Steel

V_2O_5 + 10% Na_2SO_4, at 670°C., for various periods.

III. Copper-base Alloys of Aluminium-Bronze Types

V_2O_5 + 10% Na_2SO_4, at 400° and 500°C., for various periods.

The corrosion undergone was assessed by measurement of reduction in thickness of the specimens, and by examination of the microstructure, and other observations, to determine the character of the corrosive attack.

A further section of the investigation was concerned with study of the effects of the following additive substances, when mixed with ash containing the corrodents under examination: magnesium oxide, aluminium oxide, kieselguhr (mainly silica), vermiculite, zinc oxide, and antimony trioxide. In these tests only the three high-temperature alloys were used.

The authors consider their experimental results in relation to conditions involved in commercial engine performance, and critically discuss the feasibility of minimizing corrosion by various methods. As a result of their experience in the laboratory, of results available from rig tests which are still in progress, and of the data published by others who have investigated this problem, they make the following summary of their conclusions to date:—

'Gas-turbine components are liable to be severely attacked by vanadium-bearing ash if the metal temperature is above the melting point of the ash. This attack is not likely to be serious below 650°C. Turbine fouling may occur with a variety of ashes, but is liable to be more severe with low-melting-point vanadium-bearing ones.

'None of the commercially available metals or alloys is immune from attack, but a useful degree of resistance can be obtained by the selection of suitable materials.

'Nickel-chromium alloys of the Nimonic type are more resistant than the austenitic steels, of which those containing molybdenum are the least resistant.

'Resistance to attack can be increased by the use of protective coatings high in chromium.

'Copper-base alloys, suitable for use in heat-exchangers, are attacked by vanadium-bearing ashes at temperatures considerably below the melting point of the ash.

'Dilution of the ash by the addition of magnesium oxide, zinc oxide, aluminium oxide, kieselguhr, or vermiculite, results in a considerable reduction in the rate of corrosion, and should, at the same time, minimize turbine fouling.

'Under laboratory conditions, all the above additives, with the exception of magnesium oxide, bring about a marked increase in attack when present in certain critical proportions.

'The presence of sulphur trioxide in the combustion gases will reduce the effectiveness of magnesium oxide as a corrosion inhibitor. This is unlikely to become significant at temperatures above 700°C., but will grow progressively more serious at lower temperatures.

'The use of finely divided solids which suppress the formation of sulphur trioxide may find application in protecting an additive liable to sulphation and keeping it effective, not only in the turbine but also in the cooler heat-exchanger.'

Nickel-Chromium Electrical-Resistance Alloys in Domestic Heating Elements

H. HODGES: 'Domestic Heating Elements. Trends in the Development of Nickel-Chromium Alloys.' *Electrical Rev.*, 1953, vol. 153, Sept. 18, pp. 607–12.

This article gives a review of the properties of the 80-20 nickel-chromium alloys which are so widely used in electrical-resistance heating equipment. The mechanical and physical properties characteristic of the alloys are tabulated, and particular attention is directed to the useful feature of oxide-scale adherence, which is of considerable importance in ensuring long life in the modern alloys of nickel-chromium base. Methods used for life-testing of resistance wires are described and illustrated, and attention is drawn to the increasingly severe life tests which are now used to evaluate the improved electrical-resistance alloys developed over the past 20 years. Photomicrographs demonstrate the difference in character of the scale/metal interface on 'short-life' and 'long-life' 80-20 nickel-chromium alloy wire.

Although the attack known as 'green rot' occurs only under conditions found in industrial furnaces, a short note on this subject is included, together with recommendations on methods by which such attack can be prevented.

Ceramic Coating of Nickel for High-Temperature Service

See abstract on p. 202.

Chromium-Nickel-Manganese-Molybdenum Austenitic Steels

W. E. ELLIS and M. FLEISCHMANN: 'The Substitution of Manganese for Nickel in 16-25-6 Alloy.' *Amer. Soc. Metals, Preprint 11W*, 1953; 15 pp.

This paper describes the properties of a modified form of the 16-25-6 chromium-nickel-molybdenum

steel, developed with a view to economizing in nickel content. The specification compositions of the two types of materials are shown below:—

	16–25–6 Steel %	Modified Steel %
C	0·12 max.	0·08 max.
Si	1·00 max.	1·00 max.
Mn	2·00 max.	6·50–8·50
Ni	24·00–27·00	14·00–17·00
Cr	15·00–17·00	15·00–17·50
Mo	5·50– 7·00	5·50– 7·00
N	0·10– 0·20	0·10– 0·20

The investigation reported established, for the modified steel, room-temperature properties (with and without cold work), precipitation-hardening characteristics, and microstructural data. Limited stress-rupture tests were also made, in the 1100°–1400°F. (593°–760°C.) range. In all cases comparisons were made with the properties of standard 16–25–6.

The results of the tests show that the high-manganese steel can be processed under conditions similar to those used for the original material, and that the room-temperature mechanical properties of the two types of steel are alike. In the solution-quenched condition the modified steel gave stress-rupture properties (1100°–1400°F. : 595°–760°C.) equal to those of the 16–25–6 steel, except at times beyond 1,000 hours at 1400°F. The information so far obtained suggests that the properties of the modified steel in the hot-rolled and tempered condition may be inferior to those of the 16–25–6 steel at times longer than 1,000 hours at 1200°F. (650°C.) and at higher temperatures, but this conclusion is subject to confirmation. Under all conditions, elongation at fracture was higher for the modified than for the original steel. Precipitation-hardening effects occurring in the manganese-containing steel contributed to its strength, without major sacrifice in ductility.

Shell-Moulding of Stainless Steel

W. H. DUNN and R. E. DAY: 'Shell-Moulding of Stainless Steel.' *Product Engineering*, 1953, vol. 24, Sept., pp. 129–33.

This article is written (on the background of experience built up in the foundry of Solar Aircraft Company) with a view to the needs of the engineer who, if he is to produce designs suitable for execution in the form of shell-mould castings, must have some understanding of the advantages, limitations, and potential applications of the process.

'Stainless-Steel Castings made in Shell Moulds.' *Machinery (Lond.)*, 1953, vol. 83, Oct. 23, pp. 808–9.

This short note refers to production of valves, in 18–8–3 chromium-nickel-molybdenum steel, by the shell-moulding process, as carried out in the foundry of Langley Alloys, Ltd., Slough. Typical moulds and cores are illustrated, also a representative valve body, a wedge and a bonnet, produced in shell moulds. The range of materials cast in shell moulds at this plant is shortly to be extended, to include a number of nickel-base alloys, other stainless steels, and various types of bronze.

Precipitation-Hardening in Austenitic Steels of High Carbon and Phosphorus Content

A. G. ALLTEN, J. G. Y. CHOW and A. SIMON: 'Precipitation Hardening in Austenitic Chromium-Nickel Steels containing High Carbon and Phosphorus.' *Amer. Soc. Metals, Preprint 10W*, 1953; 25 pp.

During investigation of austenitic steels for exhaust valves, in the laboratories of the Crucible Steel Company of America, it was observed that steels containing chromium 21, nickel 12, carbon 0·5, per cent., could be hardened, by solution and ageing treatment, to about Rockwell C-40. To obtain this effect it was necessary to use a very high solution temperature, but steels of this type to which about 0·3 per cent. of phosphorus was added proved susceptible to hardening when a much lower temperature was employed for the solution treatment. Since search of the literature failed to provide explanation for the effects observed, the authors undertook a systematic investigation of precipitation-hardening phenomena in such steels.

The compositions of the steels used are shown in the table on p. 215.

Three solution treatments were used: 2050°, 2150° and 2250°F. (1120°, 1175° and 1230°C.). Treatment was in each case for one hour and the specimens were water-quenched at room temperature. (It had been found that longer heating did not affect the behaviour of the steels.) Ageing was at 1100°, 1200°, 1300°, 1400°, 1500°F. (595°, 650°, 705°, 760° and 815°C.). In addition to hardness determinations, the course of precipitation was studied by means of X-ray diffraction, electrical resistivity tests and metallographic examination. The main conclusions drawn from the detailed data reported are summarized below:—

Hardnesses as high as Rockwell C-40 can be obtained in steels containing carbon 0·5, phosphorus 0·03, chromium 21, nickel 12, per cent., by treatment involving solution heating at 2250°F. (1230°C.), water-quenching, and ageing at 1300°F. (705°C.).

If the phosphorus content is raised to 0·5 per cent., and a lower temperature is used for the solution treatment, 2150°F. (1175°C.), hardnesses up to Rockwell C-46 can be produced.

Increase in phosphorus content in this type of steel lessened the time required for commencement of precipitation-hardening, and the rates of precipitation and resultant hardening were increased by such modification. Rate of softening on overageing was also increased by the higher phosphorus, due to occurrence of discontinuous precipitation.

Localized precipitation along slip lines, as well as at grain boundaries, was observed in the 0·5 per cent. carbon steels of both high and low phosphorus contents, when water-quenched from high solution

C %	Si %	Mn %	P %	Ni %	Cr %	N %
0·26	0·43	1·13	0·035	12·28	19·50	0·10
0·50	0·70	0·86	0·034	11·35	20·67	0·067
0·68	0·28	0·81	0·034	11·54	20·89	0·067
0·50	0·32	0·86	0·16	11·95	21·73	0·070
0·56	0·38	0·78	0·28	11·19	21·14	0·073
0·48	0·42	0·85	0·47	11·33	21·01	0·080

temperatures. It was established that slip occurred at temperatures below 1300°F. (705°C.) during quenching from the solution temperature.

The degree of dispersion of the precipitate found in the steels was greatly increased by an increase in phosphorus content.

No precipitate other than one having the structure of the cubic chromium carbide was found in either high- or low-phosphorus steels. Since no precipitation of a phosphorus compound was observed, an alternative mechanism is postulated as an explanation of the effects caused by phosphorus. This explanation is based on the assumption of an increase in the strain energy of the austenite lattice of the steels by interstitially dissolved phosphorus.

Effect of Heat-Treatment on the Properties of Nickel-Chromium Steel Wire

S. STORCHHEIM: 'How Heat-Treatment Affects Properties of Cold Worked 18-8 Wire.' *Iron Age*, 1953, vol. 176, Oct. 1, pp. 112-14.

This paper reports tests on the effects of heat-treatment, at various temperatures and for various times, on the magnetic, electrical and mechanical properties of commercial 18-8 chromium-nickel steel wires 0·004-in. in diameter, initially cold-drawn to a permanently magnetic condition.

Treatment at temperatures up to about 1100°F. (593°C.) had little effect on the remanence or coercive force of any of the wires. In severely cold-worked material a sudden and large increase in remanence was observed after heating at 1150°F. (620°C.), but such treatment caused little change in coercive force. After heating at higher temperatures remanence dropped and coercive force gradually rose, until a non-magnetic condition, associated with complete conversion of the magnetic martensite phase to austenite, was achieved. Sudden and large changes observed in H_c-B_r values after treatment at temperatures between 1200° and 1300°F. (648° and 704°C.) were studied in some detail and found to be associated with constitutional changes in the steels occurring within that temperature range.

It was also noted during the experiments that 'curl' of the wire resulting from drawing through the dies was lessened, though not completely removed, by treatment at 1000°F. (538°C.), showing that some degree of stress-relief resulted from heating at that temperature.

Nickel-containing Materials in Chemical Engineering: Literature Survey

'Materials of Construction for Chemical Engineering.' *7th Edition of Materials of Construction Review. Industrial and Engineering Chemistry*, 1953, vol. 45, Oct., pp. 2171-282.

This review follows broadly the lines of earlier editions, giving summaries of the literature which has appeared since the preceding survey, supplemented by tabular data including new or revised information on the properties of some metallic and non-metallic materials. The scope of the individual reviews is indicated below:—

H. O. TEEPLE: 'Nickel and High-Nickel Alloys', pp. 2215-32.

The review, supported by *270 references to original literature*, is arranged in sections covering, respectively, extraction methods and supply; general physical properties; composition and constitution of alloys: magnetic properties; high-temperature properties; fabrication (welding and brazing, mechanical forming, machining, cast alloys, and coatings); applications (high-temperature, petroleum industry, isotopes, chemical processing and miscellaneous).

W. A. LUCE: 'Stainless Steels and Other Ferrous Alloys', pp. 2241-54. This review is classified under the following headings:

Stainless Steels: corrosion, mechanical properties and structure; high-temperature ferrous alloys, welding, general.
High-Silicon Irons.
Iron-Nickel Alloys.
Austenitic Manganese Steels.

Review is based on a *bibliography* of 303 references.

'Aluminium Alloys', pp. 2175-82.
'Carbon and Graphite', pp. 2182-4.
'Cements', pp. 2185-8.
'Elastomers', pp. 2188-98.
'Fibres', pp. 2199-204.
'Iron, Mild Steels, and Low-Alloy Steels', pp. 2205-10.
'Lead and Lead Alloys', pp. 2210-14.
'Paints', pp. 2233-6.
'Plastics', pp. 2237-41.
'Tin and Its Alloys', pp. 2254-60.
'Less Common Metals', pp. 2261-9.
covering titanium, zirconium, molybdenum and tantalum.

The tabular data (pp. 2270–82) include reference information on aluminium alloys; iron, mild steel and low-alloy steels; nickel and high-nickel alloys.

Nickel-Chromium Stainless Steel in Radar Equipment

'Stainless Tubing in Radar Screens Lowers Cost, Simplifies Fabrication.' *Materials and Methods*, 1953, vol. 38, Oct., pp. 116–18.

Requirements which must be satisfied by radar scanning screens for use on naval vessels include (1) strength to resist winds, vibrations and tossing movements, (2) light weight, and (3) accurate shape, which must be retained in order to preserve operating accuracy. To meet these essential demands, a new design has been evolved. Instead of forming the supporting frame of the screen from girder sections, vertical ribs are now used, connected by tubular spanning members across the top and bottom. The steel used is A.I.S.I. 304, containing carbon 0·08 max., nickel 8–11, chromium 18–20, per cent. This article gives a step-by-step pictorial survey of the new fabricating procedure, from the tube-bending operation through to welding of the stainless-steel wire screening to the assembled frame. It is claimed that the new method gives a screen of the required lightness, strength and shape, produced more rapidly, at lower cost, and with less use of skilled labour and strategic material than was formerly required.

Photometric Determination of Titanium in Iron and Steel

W. KOCH and H. PLOUM: 'Photometric Determination of Titanium in Pig Iron and Steel (including Chromium-Nickel Steels), using Chromotropic Acid, without Separation of Iron or Alloying Elements.' *Archiv. f.d. Eisenhüttenwesen*, 1953, vol. 24, Sept.–Oct., pp. 393–6.

In photometric determination of titanium in steel the process usually employed is based on the depth of yellow-orange colouration produced by addition of hydrogen peroxide to an acidified solution of the sample. Preliminary separation of iron is usually necessary. The authors review the literature relating to this process, and refer to observations which indicated the possibility of modification of the procedure to avoid the need for preliminary removal of iron. They give particulars of research which has resulted in such improved procedure.

In acid solution, quadrivalent titanium reacts with chromotropic acid to form a complex which is characterized by a red colour, whereas trivalent iron gives a green colour, and divalent iron causes no colouration. If, therefore, the iron is reduced to the divalent state it need not be removed prior to treatment of the titanium-containing solution with chromotropic acid. Ascorbic acid is a suitable reducing medium. Preferred pH of the solution employed in the modified process is 2·5. Investigations on various types of iron, and medium- and high-alloy steels, showed that the method is suitable for pig iron, and most plain and alloy steels. If vanadium and/or tungsten are present, special calibration is required.

Butt Welding of Thin Strip of Austenitic Stainless Steel

M. S. WEINSTEIN: 'How to Butt-Weld 0·005-in. Stainless.' *Machinist*, 1953, vol. 97, Oct. 3, pp. 1653–5.

The article describes welding fixtures and technique developed (at the U.S. Naval Ordnance Laboratory, White Oak, Silver Spring, Maryland) for welding of thin strip of Type 347 (niobium-stabilized 18–8 chromium-nickel) steel in an abutted position, to simulate structural members such as T- and I- beams. By the combined use of condenser-discharge techniques, an alcohol coolant, and adequate jigging, it was found possible to weld thin stainless steel strip in an abutted position, producing joints giving consistently satisfactory results.

Corrigendum

Nickel Bulletin, Oct., 1953, p. 180.

Brit. Pat. 694,326, last patent in right-hand column. In sentence beginning 'An exchange reaction' please read as follows:

'An exchange reaction takes place between the titanium carbide *and the chromium carbide*, liberating chromium and leaving titanium carbide, which does not diffuse rapidly during the welding operation.' The italicized words were omitted from the original entry.

Binders bearing the date 195- and suitable for 1953 and 1954 Nickel Bulletins are now available. We shall be pleased to send one free of charge.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

SUBJECT INDEX TO VOLUME 26

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The individual abstracts are, in most cases, entered under the substantive titles used when they were originally reported. Leading articles are listed separately, and are also shown in the appropriate sections. In the sections they are differentiated from abstracts by an asterisk at the beginning of the title.

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